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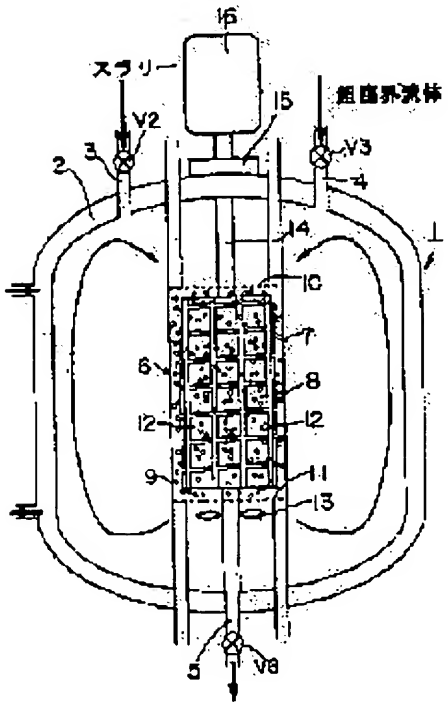
(54) METHOD AND APPARATUS FOR DISPERSING MEDIUM BY USING ULTRACRITICAL FIELD



(57)Abstract:
PROBLEM TO BE SOLVED: To easily disperse an object to be treated such as high concentration slurry by using a dispersing media.
SOLUTION: A dispersing space 6 is formed within an ultracritical vessel 1 capable of producing an ultracritical field. A dispersing media 7 and a rotating body 8 providing the dispersing media 7 with movement are provided within the dispersing space 6. The ultracritical vessel 1 is provided with a feeding port 3 for feeding an object to be treated and a feeding port 4 for feeding an ultracritical fluid. Within the ultracritical vessel 1, the object to be treated is mixed with the ultracritical fluid and passed through the dispersing space 6 in a state of reduced viscosity to be treated by dispersion. Then the mixture is drawn from a delivery port 5 of the ultracritical vessel 1 and allowed to become to ordinary temperature and atmospheric pressure. Thereby the ultracritical fluid is separated and a separated object to be treated is obtained.

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3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] The distributed space where a dispersion-medium object exercises is formed in the supercritical tub which can create a supercritical place. Supply the processed material and supercritical fluid which should be distributed in this tub, and mix supercritical fluid with this processed material, and flow into the above-mentioned distributed space and distributed processing of the mixture is carried out with the above-mentioned dispersion-medium object. The medium distribution approach using the supercritical place characterized by taking out this mixture from the above-mentioned supercritical tub, separating the above-mentioned supercritical fluid from this mixture, and obtaining the distributed processed material.

[Claim 2] The medium distribution equipment using the supercritical place formed the delivery which takes out the above-mentioned processed material by which has the supercritical tub which can create a supercritical place, formed distributed space in this tub, prepared the body of revolution which gives movement to a dispersion-medium object and this dispersion-medium object in this distributed space, and formed the feed hopper which supplies the processed material which should be distributed in this tub, and the feed hopper which supply supercritical fluid, and distributed processing was carried out with the above-mentioned dispersion-medium object, and the mixture of supercritical fluid from the inside of a tub.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention atomizes a processed material using a dispersion-medium object, and relates to the medium distribution approach and medium distribution equipment which could be made to carry out medium distribution especially of the hyperviscous processed material about the medium distribution approach and medium distribution equipment it was made to distribute in a liquid.

[0002]

[Description of the Prior Art] Although medium distribution equipment was used on the occasion of manufacture of a coating, ink, adhesives, a magnetic adjuster, etc., when atomizing processed materials, such as the slurry which contains a particle and an ultrafine particle in high concentration, i.e., a hyperviscous slurry etc., according to grinding produced between dispersion-medium objects, shear, and a grinding operation and distributing, various problems occurred and there was a case where efficient distribution became difficult.

[0003] For example, if distributed equipment is operated where a stirring aerofoil etc. is rotated by high-speed rotation in order to shorten the time amount which distribution takes, big shear generation of heat will arise. Since a processed material is hyperviscosity, efficiency of heat transfer is small, it becomes impossible to perform sufficient cooling, as a result, the solution temperature of the distributed interior of a room rises, thermal degradation is caused across a predetermined temperature requirement, and it becomes impossible to obtain a desired product, although it has cooled in the jacket etc. in order to remove this generated heat.
[0004] Moreover, since grinding of the solid particulate in a hyperviscous slurry and the crack of the floc of a solid particulate needed powerful shearing force, the large power for it had to be given for a long time and high rotation and the motor side of high torque were needed according to it, the cost of the whole equipment also became large.
[0005] Then, after distributing in the condition of having diluted with diluents, such as water and an organic solvent, in order to reduce the viscosity of a slurry in order to have distributed the hyperviscous slurry like the above conventionally for example, the approach of carrying out heating concentration of the obtained slurry, and making it into the dispersion liquid of desired concentration was taken. since [however,] this approach takes big energy in the heating concentration process of dispersion liquid, it cannot be said as an efficient process and re-condensation of a particulate material takes place in process of heating concentration -- a product -- there was a possibility of leading to degradation of description. Moreover, the approach of adding a dispersant etc. and lowering viscosity was performed, and the production cost went up with the dispersant and degradation by adding a dispersant was also produced.
[0006] Moreover, although big generation of heat like **** can be controlled if it distributes by service condition to which distributed effectiveness, such as making the filling factor of a dispersion-medium object small, and lessening the rotational frequency of a stirring aerofoil without being based on the above approaches, is reduced, if it is made such, the time amount which distribution takes will become long, and the throughput per unit time amount decreases remarkably, and cannot say that it is practical.
[0007]
[Problem(s) to be Solved by the Invention] The solution technical problem of this invention is offering the medium distribution approach and medium distribution equipment which enabled it to obtain the distributed processed material which was made to reduce viscosity in the distributed interior of a room, could process by low torque and low shear generation of heat, and was adjusted to the concentration of the request after processing even if it did not especially use a dispersant etc., when carrying out medium distribution of the processed materials, such as a hyperviscous slurry like the above.
[0008]
[Means for Solving the Problem] This invention uses the property of the supercritical fluid which can change a consistency promptly continuously from a consistency like a gas to a consistency like a liquid by changing a pressure and temperature. Supercritical fluid is mixed to processed materials, such as a hyperviscous slurry, in the supercritical tub which can create a supercritical place. The mixture is made to flow into the distributed space where the dispersion-medium object established in the tub exercises, where the viscosity of a processed material is reduced without using drugs, such as a dispersant. A medium performs grinding and distribution, the medium distribution approach using the supercritical place characterized by separating supercritical fluid from the above-mentioned mixture after distributed processing, and obtaining the distributed processed material is offered, and the above-mentioned technical problem is

solved.
[0009] In addition, in this invention, although a supercritical solvent is in a condition which is slightly less than such critical temperature besides the so-called supercritical condition in which the solvent for making a supercritical condition was meant, and a supercritical condition and supercritical fluid exceeded critical temperature and the critical pressure, and supercritical fluid, and the critical pressure, since the change of state of phase transition happens for a short time extremely, the above-mentioned supercritical condition, the subcritical state which can perform the almost same handling as supercritical fluid, and a subcritical fluid shall be included. Moreover, the actuation which produces the effectiveness of the following [blasting] is said in this invention.
(1) Crush according to the rapid cubical-expansion operation at the time of supercritical fluid permeating within between the pore or a slit, and decompressing quickly, when a dispersoid is a porous particle. Effectiveness to distribute (2) A high shear strain operation is given to a dispersoid by making the dispersion liquid of a supercritical condition blow off from the nozzle which has pore and the slit of a narrow gap with the rate of flow exceeding acoustic velocity thru/or it. Crushing, Effectiveness to distribute (3) Effectiveness which is made to collide with a wall surface etc., gives an impact operation by this to a dispersoid, and is crushed and distributed with the inertial force corresponding to the mass of the particle of a flush object.
[0010]
[Embodiment of the Invention] Drawing 1 is the explanatory view showing the configuration of the medium distribution approach by this invention, and medium distribution equipment. A supercritical tub (1) Supercritical solvents, such as a carbon dioxide, methane, ethylene, and a chlorofluorocarbon-replacing material, critical temperature, So that the supercritical place which carries out heating pressurization exceeding the critical pressure and which is made into supercritical fluid can be created About 2 times of the critical pressure, For example, it is formed in the structure of having pressure resistance to 200 atmospheric-pressure extent, and a temperature control medium is circulated around and the jacket (2) for controlling the temperature in a tub to be made at a supercritical place without spoiling the description of a dispersoid is formed.
[0011] The delivery (5) which takes out the processed material with which distributed processing of the feed hopper (3) which offers into a tub the processed material which should be distributed [slurry / high concentration], and the feed hopper (4) which supplies supercritical fluid (supercritical solvent) was formed and carried out, and the mixture of supercritical fluid from the inside of a tub is formed in the above-mentioned supercritical tub (1).
[0012] In the above-mentioned tub, the distributed space (6) for carrying out distributed processing of a processed material and the mixture of supercritical fluid with a dispersion-medium object is formed, and the body of revolution (8) which gives movement to a dispersion-medium object (7) and this dispersion-medium object is prepared in this distributed space. Although the above-mentioned distributed space provides the screen (10) of a tubed stator (9) in which the mesh, the hole, the slit, etc. were formed, and (11) so that a dispersion-medium object may not flow out up and down, you may make it surround the whole on a screen in drawing.
[0013] As the above-mentioned body of revolution (8), although various configurations are employable, the body of revolution (8) of an ANYURA-mold is used in drawing. the **** spike (12) shown in the peripheral surface of a barrel at JP,4-70050,B as this body of revolution (8) is shown in drawing -- the body of revolution (illustration abbreviation) which protrudes the body of revolution (8) which protruded ..., the body of revolution (illustration abbreviation) which

formed the protruding line so that a dispersion-medium object might be circulated covering the inside-and-outside side of tubed body of revolution, as shown in JP,3-62449,B, a pin, etc., or does not protrude -- in addition to this, proper ANYURA-mold body of revolution can be used. Moreover, it can replace with an ANYURA-mold and the body of revolution of a disk mold, a pin mold, and others can also be used. In addition, the stirring aerofoil (13) of the axial flow mold which rotates with body of revolution down the above-mentioned distributed space so that the inflow of the mixture to the above-mentioned distributed space and an outflow may be promoted is prepared.

[0014] Although the driving shaft (14) of the above-mentioned body of revolution (8) extends out of a tub through high-pressure bearing (15) and it is made to have driven it by the motor side (16), it holds this driving shaft in a tub, and you may make it drive it using a rotation field etc. from the outside of a tub.

[0015] It **, and is filled up with a glass bead, the ceramics, and the distributed medium of Steele and others in the above-mentioned distributed space (6), and processed materials, such as a high concentration slurry which should carry out opening of the bulb (V2), and should be distributed from a feed hopper (3), are prepared in the specified quantity and a supercritical tub (1). The supercritical fluid for carrying out opening of the bulb (V3), and on the other hand, forming a supercritical place from a feed hopper (4) is pressed fit in a tub, it adjusts to predetermined thermal stress, and the inside of a tub is made into a supercritical condition. After this supercritical fluid carries out heating pressurization of this solvent more than critical temperature and the critical pressure after pressing a supercritical solvent fit in a tub, is good also as supercritical fluid and considers as supercritical fluid beforehand out of a tub, you may make it press it fit in a tub. In addition, although a carbon dioxide is preferably used as a supercritical solvent, it is good to use the gaseous matter by ordinary temperature, such as methane, ethylene, and a chlorofluorocarbon-replacing material, and ordinary pressure.

[0016] Body of revolution (8) is rotated by the above-mentioned motor side (16), movement is given to the dispersion-medium object (7) in distributed space (6), and the above-mentioned processed material which flows and flows into this distributed space, and the mixture of supercritical fluid are distributed. Under the present circumstances, in order to perform cooling in a tub, although refrigerants, such as cooling water, are further circulated in body of revolution by request, this cooling is taken as a jacket and a stator, and extent that does not vanish the supercritical condition in a tub.

[0017] The mixture (dispersion liquid) distributed in the above-mentioned distributed space (6) is discharged from this distributed space (6) through a downward screen (11), and it is cooled with a jacket (2), going up near the tank wall, and through that screen (10) of the method of Gokami, this mixture goes into distributed space (6) again, and is distributed. This actuation is circulated through and repeated and distribution of the whole mixture advances.

[0018] The mixture distributed after performing predetermined time amount distribution carries out opening of the bulb (V6), and is taken out from a delivery (5) out of a tub, and by releasing to ordinary temperature and ordinary pressure, out of mixture, supercritical fluid (supercritical solvent) turns into a gas, and transpires, and a processed material can be separated and the distributed processed material, for example, a high concentration distribution slurry, can be obtained. May lead to a blasting tub, may give an impact operation so that a postscript may be carried out, and may promote distribution, the feed hopper (3) of the above-mentioned supercritical tub (1) is made to circulate through the processed material taken [above-mentioned] out picking again, and you may make it repeat the above-mentioned actuation in the case of the

above-mentioned ejection.
[0019] One example of the distributed system which enforces the above-mentioned distributed approach is shown in drawing 2 . In drawing, preliminary mixing equipments, such as kneaders (17), such as a roll mill and a kneader, and a planetary mixer (18), are formed so that a request can perform preliminary mixing, dispersoids, such as a particle and an ultrafine particle, a solvent, a dispersant, etc. are mixed with this preliminary mixing equipment, and this mixture is supplied to the distributed sample adjustment tank (19) with pumps (P1), such as Sneh-KUPOMPU and a screw extruder. By this adjustment tank (19), required feed to pulverizing machine, drugs, a solvent, etc. are mixed, and the agitator (20) is formed so that precipitation of a particle, and condensation and separation of a distributed solute and a solvent may be prevented, as preferably shown in drawing. In this case, what is necessary is just to use, when wished especially although drugs, such as a dispersant, are not necessarily required.
[0020] The above-mentioned tank (20) A bulb (V1), a distributed sample liquid-sending pump (P2), Connect with the feed hopper (3) of a supercritical tub (1) through a bulb (V2), and temperature control of this supercritical tub (1) is carried out with a jacket with a temperature control (2). It consists of feed hoppers (4) of another side so that supercritical fluid may be supplied. It connects with the above-mentioned bulb (V3) through the compressor-pump for pressurization (P5) for Rhine which leads to the source of supply of supercritical fluid (supercritical solvent) to make a bulb (V4), a filter (F1), and a supercritical place.
[0021] Rhine with a heater which a pressure gage (G) and a thermometer (T1) are formed in the above-mentioned supercritical tub (1), heats at an external heater in a delivery (5), and prevented supercooling -- (21) connects -- having -- this Rhine -- (21) is connected with the blasting tub (22) through a reducing valve with an actuator (V6).
[0022] Although the dashboard (23) was formed up, above-mentioned Rhine is connected to exhaust nozzles (24), such as a blasting nozzle and a blasting aperture, a collision plate with an enclosure (25) is formed ahead of exhaust nozzles (24), such as this blasting nozzle and a blasting aperture, and the impact operation is given into the above-mentioned blasting tub (22), an exhaust nozzle is made to counter and it may be made to carry out a counterflow collision. In addition, the above-mentioned blasting nozzle etc. can also promote distribution by shearing by using the nozzle with a **** heater used for the particle manufacture process of having used supercritical fluid, having prevented plugging by freezing, and using the capillary tube of a proper tube diameter for above-mentioned Rhine (21), or establishing the parallel tabular passage of a proper gap in it on the way.
[0023] A buffer tub (26) is connected to the above-mentioned blasting tub (22) through a filter (F2) and the compressor-pump for pressurization (P4) so that the supercritical solvents separated from dispersion liquid may be collected, and this buffer tub (26) is connected to the above-mentioned pump (P5) through the bulb (V5). In addition, - (V5) is good to use stop valves, such as a ball valve with the above-mentioned bulb (V1) and an actuator, and, as for the above-mentioned filter (F1) etc., a metal sintering porous body, a ceramic, etc. are used (F2).
[0024] A tank (degassing tub) (27) is connected to the lower part of the above-mentioned blasting tub (22) through a liquid-sending pump (P3) and a flowmeter (M4), and this tank (27) is heated in a temperature-control addition heat jacket (28), and has been made to carry out stirring mixing of the dispersion liquid with the agitator (29). The recovery system which is open for free passage to the above-mentioned buffer tub (26) so that the supercritical solvents which are not collected [which has formed the thermometer (T2) in this tank (27), and were separated above this tank (27) from dispersion liquid by request] may be collected may be prepared.

[0025] In addition, the exhaust port with a bulb (30) for discharging a penetrant remover, respectively to the above-mentioned tank for distributed sample adjustment (19), a supercritical tub (1), a blasting tub (22), and a tank (27), (31), (32), and (33) It is prepared. Moreover, data processing is sent and carried out to a computer, a signal is sent to the above-mentioned pump, the actuator of a bulb, the temperature controller of a heating jacket, the heater of Rhine, etc., and the above-mentioned thermometer (T1), the temperature acquired from (T2), the pressure obtained from a manometer (G), the flow rate data obtained from a flow meter control the amount of heating of the amount of liquid sending of a pump, closing motion of a bulb, a jacket, and a heater etc., respectively.

[0026] Moreover, what is necessary is to form the circulation line (34) which connects the above-mentioned blasting tub (22) and the above-mentioned feed hopper (3), to form a bulb (V7) and a pump (P6) in this Rhine, and just to make it repeat actuation of blasting by the distribution in the supercritical place describing above, and the blasting tub etc., when making this supercritical tub (1) circulate through the dispersion liquid (processed material) taken out from the above-mentioned supercritical tub (1).

[0027]

[Effect of the Invention] This invention is constituted as mentioned above and mixes processed materials, such as a high-concentration high viscosity slurry, with supercritical fluid in a supercritical tub. Since it was made to carry out distributed processing with the dispersion-medium object which the distributed space in which the mixture was formed in this tub is circulated, and exercises in this distributed space Viscosity is reduced by mixing with supercritical fluid, even if it is the processed material of high viscosity. If distributing enough is possible, the above-mentioned mixture after distributed processing is taken out from a tub and it releases to atmospheric pressure even if it can make a dispersion-medium object rotate the body of revolution which gives movement with the driving force of low torque and does not use a dispersant It can become a gas, can transpire, and can dissociate from a processed material, and the above-mentioned supercritical fluid can obtain products, such as a high concentration distribution slurry to which concentration was raised.

[Translation done.]

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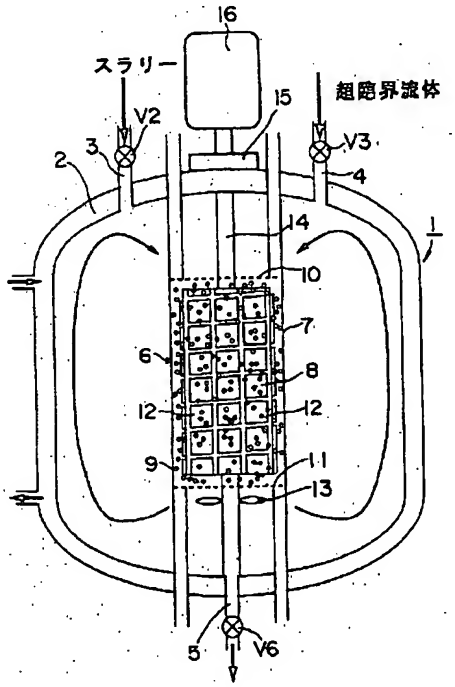
(54)【発明の名称】　超臨界場を用いた媒体分散方法及び媒体分散装置

(57)【要約】

【課題】　高濃度スラリー等の被処理物を分散媒体を用いて容易に分散できるようにする。

【解決手段】　超臨界場を作成できる超臨界槽（１）内に分散空間（６）を形成し、この分散空間（６）内に分散媒体（７）と該分散媒体（７）に運動を与える回転体（８）が設けられている。上記槽には被処理物を供給する供給口（３）と超臨界流体を供給する供給口（４）がある。槽内において、上記被処理物は超臨界流体と混合され、粘度が低減された状態で上記分散空間を流通して分散処理される。その後、上記混合物は超臨界槽（１）の吐出口（５）から取り出され、常温、常圧にすることにより上記超臨界流体は分離され、分散された被処理物が得られる。



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【特許請求の範囲】

【請求項１】　超臨界場を作成できる超臨界槽内に分散媒体が運動する分散空間を形成し、該槽内に分散すべき被処理物と超臨界流体を供給し、該被処理物と超臨界流体を混合しその混合物を上記分散空間に流入して上記分散媒体により分散処理し、該混合物を上記超臨界槽から取り出し、該混合物から上記超臨界流体を分離し、分散された被処理物を得ることを特徴とする超臨界場を用いた媒体分散方法。

【請求項２】　超臨界場を作成できる超臨界槽を有し、該槽内に分散空間を形成し、該分散空間内に分散媒体と該分散媒体に運動を与える回転体を設け、該槽内に分散すべき被処理物を供給する供給口と超臨界流体を供給する供給口を形成し、かつ上記分散媒体により分散処理された上記被処理物と超臨界流体の混合物を槽内から取り出す吐出口を形成した超臨界場を用いる媒体分散装置。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、分散媒体を用いて被処理物を微粒子化し、液体中に分散するようにした媒体分散方法及び媒体分散装置に関し、特に高粘度の被処理物を媒体分散できるようにした媒体分散方法及び媒体分散装置に係るものである。

【０００２】

【従来の技術】塗料、インキ、接着剤、磁気材料等の製造に際し、媒体分散装置が用いられているが、高濃度に微粒子や超微粒子を含むスラリー、すなわち高粘度スラリー等の被処理物を分散媒体間に生じる粉砕、せん断、摩砕作用により微粒子化して分散する場合、種々の問題が発生し、効率的な分散が困難となる場合があった。

【０００３】例えば、分散に要する時間を短縮させるために高速回転で撹拌翼等を回転させた状態で分散装置を運転すると、大きなせん断発熱が生じる。この発生した熱を除去するため、ジャケット等で冷却しているが、被処理物が高粘度であるので、伝熱効率が小さく、充分な除熱が行えなくなり、その結果分散室内の液温が上昇し、所定の温度範囲を超えて熱的劣化を来し、所望の製品を得られなくなる。

【０００４】また、高粘度スラリー中の固体粒子の粉砕や固体粒子の凝集体の解砕は、強力なせん断力を必要とし、そのための大動力を長時間与えなければならず、それに応じて高回転、高トルクのモーターを必要とするから、装置全体のコストも大きくなった。

【０００５】そこで、従来上記の如き高粘度スラリーの分散を行うには、例えばスラリーの粘度を低減させるために、水、有機溶媒等の希釈液で希釈した状態で分散を行ったのち、得られたスラリーを加熱濃縮し、所望の濃度の分散液とする方法が採られていた。しかし、この方法では分散液の加熱濃縮過程で大きなエネルギーを要し、効率的なプロセスとはいえないし、加熱濃縮の過程で分散粒子の再凝集が起こるため、製品性状の劣化につながるおそれがあった。また、分散剤等を添加して粘度を下げる方法も行われているが、分散剤により生産コストが上昇し、分散剤を添加することによる劣化も生じていた。

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【０００６】また、上記のような方法によらないで、分散媒体の充填率を小さくし、撹拌翼の回転数を少なくする等分散効率を低下させるような運転条件で分散すれば、上述の如き大きな発熱を抑制することができるが、そのようにすると分散に要する時間が長くなり、単位時間当りの処理量が著しく少なくなって、実用的とは言えない。

【０００７】

【発明の解決課題】本発明の解決課題は、上記の如き高粘度スラリー等の被処理物を媒体分散する際、特に分散剤等を用いなくても、分散室内では粘度を低減させて低トルク、低せん断発熱で処理でき、処理後所望の濃度に調整された分散された被処理物を得られるようにした媒体分散方法及び媒体分散装置を提供することである。

【０００８】

【課題解決の手段】本発明は、圧力や温度を変化させることにより気体のような密度から液体のような密度まで密度を連続的に速やかに変えることができる超臨界流体の性質を利用し、超臨界場を作成できる超臨界槽内において高粘度スラリー等の被処理物に超臨界流体を混合し、被処理物の粘度を分散剤等の薬剤を用いることなく低減させた状態でその混合物を槽内に設けた分散媒体が運動する分散空間に流入させ、媒体により粉砕、分散を行い、分散処理後上記混合物から超臨界流体を分離し、分散された被処理物を得ることを特徴とする超臨界場を用いた媒体分散方法が提供され、上記課題が解決される。

【０００９】なお、本発明において、超臨界溶媒とは、超臨界状態を作るための溶媒を意味し、また超臨界状態、超臨界流体とは臨界温度、臨界圧力を超えたいわゆる超臨界状態、超臨界流体の他、そのような臨界温度、臨界圧力をわずかに下回るような状態ではあるが相転移の状態変化が極めて短時間に起こるため上記超臨界状態、超臨界流体とほぼ同様の取り扱いができるような亜臨界状態、亜臨界流体を含むものとする。また、本発明において爆砕とは下記の効果を生ずる操作をいう。

（１）　分散質が多孔性粒子である場合、その細孔や細隙間内に超臨界流体が浸入し急速に減圧する際の急激な体積膨張作用により破砕、分散する効果、（２）　細孔や狭い間隙のスリットを有するノズルから超臨界状態の分散液を音速ないしそれを超える流速をもって噴出させることにより分散質に高剪断変形作用を与えて破砕、分散する効果、（３）　噴出流体の微粒子の質量に対応する慣性力によって壁面等に衝突させ、これにより分散質に衝撃作用を与えて破砕、分散する効果。

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【0010】

【発明の実施の形態】図1は、本発明による媒体分散方法及び媒体分散装置の構成を示す説明図であり、超臨界槽（1）は、二酸化炭素、メタン、エチレン、代替フロン等の超臨界溶媒を臨界温度、臨界圧力を超えて加熱加圧し超臨界流体にする超臨界場を作成することができるよう臨界圧力の2倍程度、例えば200気圧程度まで耐圧性を有する構造に形成され、周囲に調温媒体を流通させて槽内の温度を分散質の性状を損なわないで超臨界場にできるようにコントロールするためのジャケット（2）が形成されている。

【0011】上記超臨界槽（1）には、高濃度スラリー等の分散すべき被処理物を槽内に供する供給口（3）と、超臨界流体（超臨界溶媒）を供給する供給口（4）が形成され、かつ分散処理された被処理物と超臨界流体の混合物を槽内から取り出す吐出口（5）が形成されている。

【0012】上記槽内には、分散媒体により被処理物と超臨界流体の混合物を分散処理するための分散空間（6）が形成され、該分散空間内には分散媒体（7）と該分散媒体に運動を与える回転体（8）が設けられている。図において、上記分散空間は筒状のステーター（9）の上下に分散媒体が流出しないよう網目、孔、スリット等を形成したスクリーン(10)，(11)を設けてあるが、全体をスクリーンで囲むようにしてもよい。

【0013】上記回転体（8）としては、種々の構成を採用することができるが、図においてはアニュラー型の回転体（8）を用いている。該回転体（8）は、図に示すように筒体の周面に特公平4－70050号公報に示す如きスパイク(12)・・・を突設した回転体（8）や、特公平3－62449号に示すように分散媒体を筒状の回転体の内外面にわたって循環させるよう突条を形成した回転体（図示略）、ピン等を突設し若しくは突設していない回転体（図示略）その他適宜のアニュラー型回転体を用いることができる。また、アニュラー型に代えてディスク型、ピン型その他の回転体を用いることもできる。なお、上記分散空間への混合物の流入、流出を促進するよう上記分散空間の下方には回転体と共に回転する軸流型の撹拌翼(13)を設けてある。

【0014】上記回転体（8）の駆動軸(14)は、高圧軸受(15)を介して槽外に延出し、モーター(16)により駆動するようにしてあるが、該駆動軸を槽内に保持して槽外から回転移動磁界等を用いて駆動するようにしてもよい。

【0015】而して、上記分散空間（6）内にガラスビーズ、セラミックス、スチールその他の分散媒体を充填し、バルブ(V2)を開口して供給口（3）から分散すべき高濃度スラリー等の被処理物を所定量、超臨界槽（1）内に仕込む。一方、バルブ(V3)を開口して供給口（4）から超臨界場を形成するための超臨界流体を槽内に圧入し、所定の温度圧力に調整して槽内を超臨界状態とする。この超臨界流体は、槽内に超臨界溶媒を圧入してから該溶媒を臨界温度、臨界圧力以上に加熱加圧して超臨界流体としてもよいし、槽外で予め超臨界流体としてから槽内に圧入するようにしてもよい。なお、超臨界溶媒としては、好ましくは二酸化炭素が用いられるが、その他メタン、エチレン、代替フロン等常温、常圧で気体の物質を用いるとよい。

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【0016】上記モーター(16)により回転体（8）を回転し、分散空間（6）内の分散媒体（7）に運動を与え、該分散空間に流入、流出する上記被処理物と超臨界流体の混合物を分散する。この際、槽内の除熱を行うため、ジャケット及びステーター、さらに所望により回転体内に冷却水等の冷媒を流通させるが、この除熱は槽内の超臨界状態を消失させない程度とする。

【0017】上記分散空間（6）内で分散された混合物（分散液）は、下方のスクリーン(11)を通って該分散空間（6）から排出され、この混合物は槽壁近傍を上昇しながらジャケット（2）により除熱され、その後上方のスクリーン(10)を通って再び分散空間（6）に入り、分散される。この操作は、循環して繰り返され、混合物全体の分散が進行する。

【0018】所定の時間分散を行った後、分散された混合物はバルブ(V6)を開口して吐出口（5）から槽外に取り出され、常温、常圧に解放することにより混合物中から超臨界流体（超臨界溶媒）が気体となって蒸散し被処理物が分離され、分散された被処理物、例えば高濃度分散スラリーを得ることができる。上記取り出しの際、後記するように爆砕槽に導いて衝撃作用を与え、分散を促進させてもよいし、上記取り出された被処理物を再び上記超臨界槽（1）の供給口（3）に循環させて上記操作を繰り返すようにしてもよい。

【0019】上記分散方法を実施する分散システムの一実施例が、図2に示されている。図において、所望により予備混合を行うことができるようロールミル、ニーダー等の捏和機(17)やプラネタリーミキサー(18)等の予備混合装置が設けられ、該予備混合装置により微粒子、超微粒子等の分散質、溶媒、分散剤等を混合し、この混合物をスネークポンプ、スクリュー押出機等のポンプ(P1)により分散試料調整タンク(19)に供給するようにしてある。該調整タンク(19)では、必要な砕料、薬剤、溶媒等が混合され、好ましくは図に示すように粒子の沈澱、凝集や分散溶質と溶媒の分離を防ぐよう撹拌機(20)が設けられている。この場合、分散剤等の薬剤は必ずしも必要でないが、特に望まれるときに用いればよい。

【0020】上記タンク(20)は、バルブ(V1)、分散試料送液ポンプ(P2)、バルブ(V2)を介し超臨界槽（1）の供給口（3）に連結し、該超臨界槽（1）は温度コントロール付のジャケット（2）により調温され、他方の供給口（4）からは超臨界流体を供給するよう構成されてお

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り、超臨界流体（超臨界溶媒）の供給源に通じるラインがバルブ(V4)、フィルター(F1)、超臨界場を作るための加圧用コンプレッサーポンプ(P5)を介して上記バルブ(V3)に接続されている。

【００２１】上記超臨界槽（１）には圧力計（Ｇ）、温度計(T1)が設けられ、吐出口（５）には外部ヒーターにより加熱し、過冷却を防止するようにしたヒーター付ライン(21)が接続され、該ライン(21)はアクチュエーター付減圧バルブ(V6)を介し爆砕槽(22)に連結している。

【００２２】上記爆砕槽(22)内には、上方に仕切板(23)が設けられ、上記ラインは爆砕ノズル、爆砕窓等の噴出口(24)に接続しており、該爆砕ノズル、爆砕窓等の噴出口(24)の前方には囲い付衝突板(25)を形成して衝撃作用を与えるようにしてあるが、噴出口を対向させて向流衝突させるようにしてもよい。なお、上記爆砕ノズル等は、超臨界流体を用いた微粒子製造プロセスに用いる如きヒーター付ノズルを使用し、凍結による詰まりを防止するようにしてあり、また上記ライン(21)に適宜な管径の毛細管を用いたり、途中に適宜の間隙の平行板状の流路を設けることにより、せん断作用による分散を促進させることもできる。

【００２３】上記爆砕槽(22)には、分散液から分離した超臨界溶媒を回収するようフィルター(F2)、加圧用コンプレッサーポンプ(P4)を介し緩衝槽(26)が接続され、該緩衝槽(26)はバルブ(V5)を介し上記ポンプ(P5)に接続されている。なお、上記バルブ(V1)～(V5)は、アクチュエーター付ボールバルブ等のストップバルブを用いるとよく、また上記フィルター(F1)，(F2)等は金属焼結多孔体、セラミック等が用いられる。

【００２４】上記爆砕槽(22)の下部には送液ポンプ(P3)、流量計(M4)を介して貯槽（脱泡槽）(27)が接続され、該貯槽(27)は温度コントロール付加熱ジャケット(28)で加熱され、撹拌機(29)で分散液を撹拌混合するようにしてある。該貯槽(27)には温度計(T2)を設けてあり、また所望により該貯槽(27)の上方に分散液から分離した未回収の超臨界溶媒を回収するよう上記緩衝槽(26)に連通する回収装置を設けてもよい。

【００２５】なお、上記分散試料調整用タンク(19)、超臨界槽（１）、爆砕槽(22)、貯槽(27)にはそれぞれ洗浄液を排出するためのバルブ付排出口(30)，(31)，(32)，(33) が設けられている。また、上記温度計(T1)，(T2)から得られる温度、圧力計（Ｇ)から得られる圧力、流量

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計から得られる流量データ等は、コンピューターに送られ、演算処理され、上記ポンプ、バルブのアクチュエーター、加熱ジャケットの温度コントローラー、ラインのヒーター等に信号が送られ、それぞれポンプの送液量、バルブの開閉、ジャケットおよびヒーターの加熱量等を制御する。

【００２６】また、上記超臨界槽（１）から取り出した分散液（被処理物）を該超臨界槽（１）に循環させる場合は、上記爆砕槽(22)と上記供給口（３）を連絡する循環ライン(34)を形成し、該ラインにバルブ(V7)、ポンプ(P6)を設けて上記超臨界場における分散、爆砕槽による爆砕等の操作を繰り返すようにすればよい。

【００２７】

【発明の効果】本発明は上記のように構成され、高濃度の高粘性スラリー等の被処理物を超臨界槽内において超臨界流体と混合し、その混合物を該槽内に形成した分散空間に流通させ、該分散空間内で運動する分散媒体により分散処理するようにしたので、高粘性の被処理物であっても超臨界流体と混合することにより粘度が低減され、分散媒体に運動を与える回転体を低トルクの駆動力で回転させることができ、分散剤を用いなくても充分分散することが可能であり、分散処理後上記混合物を槽から取り出し、大気圧に解放すると、上記超臨界流体は気体となって蒸散して被処理物から分離し、濃度が高められた高濃度分散スラリー等の製品を得ることができる。

【図面の簡単な説明】

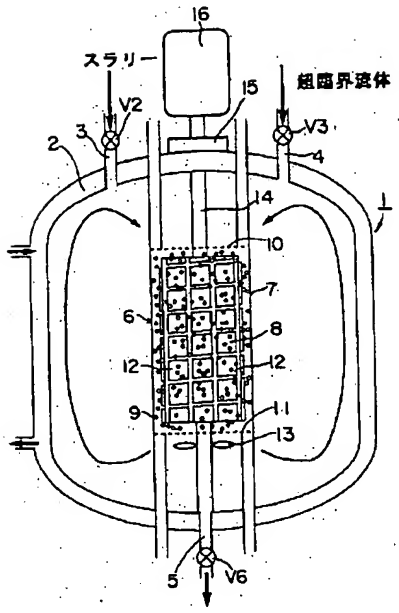
【図１】本発明の構成の一実施例を示す説明図。

【図２】本発明の分散システムの一実施例を示す説明図。

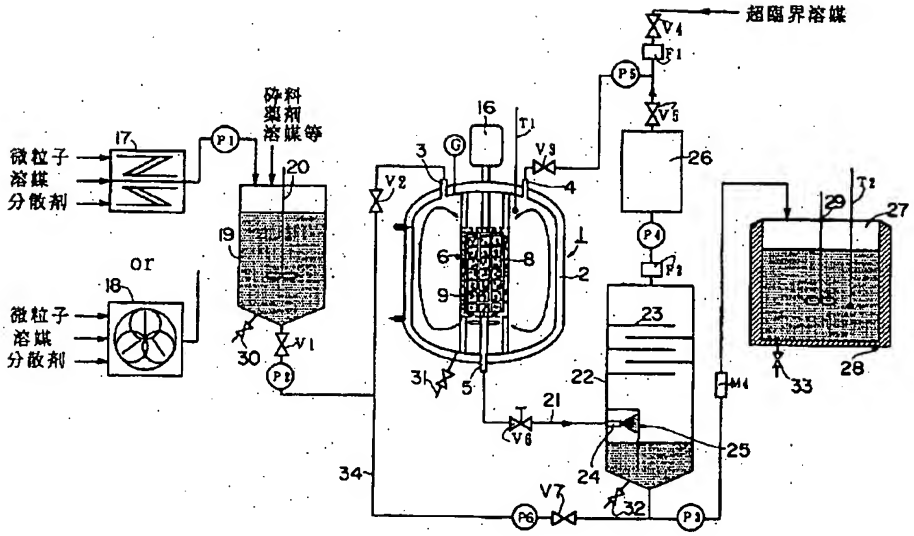
【符号の説明】

1　超臨界槽内
2　ジャケット
3　供給口
4　供給口
5　吐出口
6　分散空間
7　分散媒体
8　回転体
19　分散試料調整タンク
22　爆砕槽
26　緩衝槽
27　貯槽

【図1】


16
スラリー
超臨界流体
15
V2
V3
3
2
4
14
10
7
6
8
12
12
9
11
13
5
V6

【図2】


超臨界溶媒
V4
P1
P5
V5
26
16
T1
3
G
V3
V2
4
6
8
9
2
P4
P2
23
29
T2
27
22
M1
33
28
31
5
21
V8
25
24
34
V7
32
P6
P3
17
P1
砂料
薬剤
溶媒等
20
微粒子
溶媒
分散剤
19
or
18
微粒子
溶媒
分散剤
30
V1
P2